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FRICTION AT HIGH SLIDING VELOCITIES OF OXIDE FILMS ON STEEL SURFACES BOUNDARY-LUBRICATED WITH STEARIC-ACID SOLUTIONS

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SUMMARY

Experiments were conducted to establish the capabilities of a fatty acid (stearic) as an additive for lubrication of steel surfaces at high sliding velocities (75 to 7000 ft/min) with severe loading (169 to 1543 grams, initial Hertz surface stress, 108,000 to 194,000 lb/sq in.) and to determine the efficacy of prepared oxide films in this type of lubrication.

It was found that lubrication with stearic acid as an additive in cetane was effective at sliding velocities up to 3000 feet per minute both for clean steel surfaces and for surfaces coated with ferric oxide Fe_2O_3 (1000 A thick). A prepared film of ferroso-ferric oxide Fe_3O_4 (1000 A thick) prevented lubrication failure with 0.5-percent stearic acid in cetane at sliding velocities higher than 7000 feet per minute (269 gram load) and with loads to 769 grams at a sliding velocity of 6000 feet per minute. Lubrication failures were probably caused by melting of the metallic soap formed by interaction of the stearic acid with the steel surfaces. The experiments indicate that the type of surface oxide and the thickness of the oxide film are important in determining the effectiveness of stearic acid as an additive in lubrication at high sliding velocities.

INTRODUCTION

The use of low-viscosity oils in aircraft engines to allow satisfactory operation at low temperatures has resulted in marginal boundary lubrication at operating temperatures (reference 1). The use of lubricant additives for providing increased load capacity of sliding surfaces is being considered. References 2 and 3 point

out that with a temperature-active (extreme-pressure) type of additive lubrication may be ineffective at high sliding velocities because an effective chemical reaction between the surfaces and the additives to provide a lubricating film may not have time to occur.

Studies made at relatively low temperatures (references 4 to 9) have established the point that, with a fatty-acid type of additive, high contact temperatures or pressures are unnecessary for formation of an effective lubricating film. Strongly adsorbed, oriented molecules of the fatty acids are deposited on the metallic surfaces with which they react to form metallic soap. Because a preformed lubricating film is available, a fatty-acid type of additive would offer greater possibilities for lubrication at high sliding velocities than would the temperature-active (extreme-pressure) type of additive, which must form its lubricating film under contact conditions. Fatty acids are, however, generally considered to provide less load-carrying capacity than is obtainable from temperature-active materials. Furthermore, fatty acids are not considered as effective at high temperatures (reference 10).

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Fundamental corrosion studies reported in reference 7 indicate the necessity for surface oxides or moisture in the chemical action of fatty acids with several metals. Lubrication studies reported in references 4, 8, and 9 show the importance of moisture and oxides in lubrication by fatty acids. No data have been published, however, to show the effects of different oxides of iron on the lubrication process with fatty acids.

The object of the research reported herein was to investigate a fatty acid (stearic acid) as an additive for lubrication of steel at high sliding velocities and to determine the effect of prepared surface oxide films on lubrication by fatty acids. Experiments were conducted at the NACA Lewis laboratory using a sliding-friction apparatus that allowed friction to be measured and boundary lubrication effectiveness to be determined at high sliding velocities (50 to 18,000 ft/min) with high surface loads (initial Hertz surface stresses, 108,000 to 194,000 lb/sq in.). Friction specimens were used with prepared films of ferroso-ferric oxide Fe_3O_4 and ferric oxide Fe_2O_3 and with clean steel surfaces.

APPARATUS AND PROCEDURE

Friction apparatus. - The friction apparatus used for these experiments is that described in reference 11. A diagrammatic

sketch of the basic parts of the apparatus is presented in figure 1. The principal elements of the apparatus were the specimens, which were an elastically restrained spherical rider and a rotating disk. The disk specimens had an outer diameter of 13 inches and were made of normalized SAE 1020 steel, Rockwell A-50. The rider specimens used were commercial balls, 1/4 inch in diameter, and were made of SAE 1095 steel hardened to Rockwell C-60. The rider load P was applied along the vertical axis of the rider holder. Friction-force readings F were obtained from an indicating-type calibrated potentiometer connected to a strain-gage - dynamometer-ring assembly and were recorded using a motion-picture camera (64 frames/sec) timed to operate for the 3 seconds covering the duration of each friction run. The coefficient of kinetic friction μ_k was computed from the equation

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$$\mu_k = \frac{F}{P}$$

where F is the measured friction force and P is the applied normal load.

A motor-driven radial-feed mechanism, calibrated to indicate radial position of the rider, caused the rider to traverse a spiral path on the rotating disk so that portions of the wear track did not overlap. The disk was mounted on a flywheel that was driven through a flexible coupling by a hydraulic motor operating under constant fluid pressure with speed adjusted by varying the flow of the hydraulic fluid; this arrangement allowed good speed control over a range of sliding velocities between 50 and 18,000 feet per minute. The disk and the rider were enclosed, permitting the pre-operating atmosphere of dried air to be slightly pressurized so that the contaminated room atmosphere did not contact the disk prior to the application of the lubricant. In order to prevent excessive evaporation of the lubricants, dried air was not allowed to flow over the specimens during the experiments.

Film-formation apparatus. - The oxide films were formed by heating the steel disks in air in a vacuum furnace at either atmospheric or reduced pressures. This vacuum furnace, shown in figure 2, consisted of a 2000-watt circular heater built to accommodate the disk specimens mounted inside a 15-inch metal bell jar. The specimen and the heater were completely enclosed by a reflective aluminum shield to aid in obtaining uniform heating. The chamber was connected to vacuum pumps with an air bleed into the pumping system that allowed very close control of the pressure inside the chamber.

Specimen preparation. - The disks were finished and cleaned according to the following procedure (described in greater detail in reference 11): The disks were finished by surface grinding and nondirectional lapping to produce a surface having random finishing marks with a roughness of 4 to 8 microinches rms as measured with a profilometer. The cleaning process included in sequence: soaking and wiping in a low aromatic cleaning naptha, wiping with clean cloths saturated with a solution containing equal parts of acetone and benzene, scrubbing with moist levigated alumina powder, rinsing under tap water to remove the alumina, testing for cleanness by observing if water will wet the surface, immersing in redistilled 95-percent ethyl alcohol to remove the water, rinsing with additional redistilled 95-percent ethyl alcohol, and drying on the friction apparatus in an atmosphere of dried and filtered air. The rider specimens were cleaned by wiping with a cloth saturated with ethyl alcohol and by rinsing with redistilled 95-percent ethyl alcohol. The rider was allowed to dry on the apparatus.

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The films of Fe_3O_4 were formed on the disks by heating in a restricted oxygen supply. The disks were heated at a pressure of 10 millimeters of mercury (absolute) and a temperature of approximately 875° F for a period of time sufficient to form a film of thickness that produced a blue interference color. The color of the film indicated that its thickness was approximately 1000 angstrom units. The disks were allowed to cool to room temperature in the furnace while maintaining the pressure at 10 millimeters of mercury over a long period of time. The Fe_2O_3 films were formed on the disk specimens by heating at atmospheric pressure and a temperature of 875° F for a period of time sufficient to produce a blue film, indicating that the oxide thickness was approximately 1000 angstrom units. The chemical composition and crystalline characteristics of each film were determined by electron-diffraction patterns obtained from small steel specimens prepared at the same time the friction disks were prepared. These specimens were placed on the inner edge of each disk and subjected to the same treatment as the disk. Figure 3 shows representative patterns obtained from the film materials used in these experiments.

The lubricants used in these experiments were mixtures of stearic acid in cetane or stearic acid. The stearic acid used was of the highest purity available from commercial sources and it was used at various weight concentrations in cetane as the base carrier. The cetane was stored in a dark room to prevent promotion of its oxidation by light and was percolated through Fuller's earth, alumina, and silica gel immediately prior to its use in every experiment in order to remove any extraneous materials. The lubricant solutions

were made immediately prior to each friction experiment. The lubricants were handled at all times in glassware cleaned in a chemical cleaning solution (chromic-acid type) and were deposited on the disk specimen by drops from a clean platinum dipper. After the lubricant had spread over the whole surface, the disk was rotated at the maximum speed of the experiment (approximately 2500 rpm) for 5 minutes causing the excess lubricant to be thrown off leaving only a very thin film. All the experiments were completed within 1 hour of deposition of the film; previous experience indicated that evaporation of the lubricant did not affect the friction results within that period of time. In the experiments using pure stearic acid, the material was deposited on the disks, which had been heated (reheated in the case of oxide films) to a temperature just above the melting point (157° F) of the stearic acid. The disk was rotated and the stearic acid was rubbed on the disk surface with lens tissue using a circumferential motion to obtain a very thin film. Reference 6 indicates that a film applied in this manner is oriented and approaches a monomolecular layer of the lubricant on the points of the surface asperities.

Experimental procedure. - During the experiments the disk was rotated at a predetermined speed. By means of a cam arrangement, the loaded rider was lowered onto the disk as the radial feed was started. As the rider traversed the disk, friction force, as indicated by the potentiometer, was observed and photographically recorded and disk rotative speed was determined with an electric revolution counter and synchronized timer. The timer controlled the operation cycle of the camera, the radial traverse mechanism, and the revolution counter. The runs were terminated by lifting the rider from the disk surface. Mean sliding velocity for the runs was computed from the recorded rotative disk speed and the mean diameter of the rider path. Change in diameter of the rider path on the disk during radial travel of the rider caused a maximum deviation in sliding velocity of approximately 3 percent from the mean value. An unworn area of a rider was used in each run. Experiments were conducted under relatively constant conditions of room temperature (74° F) and room humidity (40 percent). The films and the surfaces of the disk specimen were studied using common physical measurement and metallographic equipment before and after the experiments. Hardness and surface-roughness measurements were used as control factors in evaluation of the disk specimens prior to the experiments. The electron-diffraction patterns were produced with the diffraction adapter of a type EMB-4 RCA electron microscope.

Basis for interpretation of data. - The friction data presented are complete for a representative experiment of each lubrication variable studied and were selected from a mass of data from several experiments on each variable. In all but isolated cases, the maximum experimental error in friction coefficient for a given condition of lubrication was within ± 0.02. When unstable friction values were obtained, it was most generally under conditions where marginal lubrication prevailed. Effective boundary lubrication may be defined as a nonhydrodynamic condition of sliding of two mating surfaces under which friction is consistently low (approximately 0.10) with no surface welding. For comparative purposes, a load of 269 grams was used for the majority of the curves presented; this load produces an initial Hertz surface stress (126,000 lb/sq in.) that is in the range of stresses commonly attained in aircraft-engine components that require lubrication (reference 12).

RESULTS AND DISCUSSION

The following two parameters have been investigated in order to determine their effect on the friction properties of steel surfaces lubricated with stearic acid: (1) lubrication with various concentrations, and (2) effect of surface-oxide treatment.

Lubrication with Various Concentrations

Clean steel. - The effect of sliding velocity on friction is shown in figure 4 for clean steel surfaces lubricated with solutions of stearic acid and cetane, for dry steel surfaces, for surfaces lubricated with pure cetane, and for surfaces lubricated with pure stearic acid. The curve for dry steel is taken from reference 11 and the curve for pure cetane on steel is taken from reference 2. The clean steel refers to a surface having a film which forms during normal handling that is predominantly Fe_3O_4 and is approximately 20 angstrom units thick. All nominally clean steel surfaces handled in air have such an oxide film.

As indicated in figure 4, there is no appreciable effect of the differences in concentrations of stearic acid investigated on friction at sliding velocities below approximately 3000 feet per minute. The curves presented for 0.1-percent and 0.5-percent stearic acid in cetane are nearly coincident in that range of sliding velocities. At the higher sliding velocities, the effect of additive concentration becomes more evident as partial-lubrication failures occur. Surface welding and a change in the

slope of the curves occurred simultaneously, indicating the presence of a critical sliding velocity. When the factors of friction and surface welding as related to changes in surface temperature are considered, it seems (and will be discussed later in connection with lubrication mechanism) that the mechanism of failure of the lubrication provided by the stearic acid in cetane solutions is primarily one of melting or softening of the metallic soap, allowing contact of clean metallic asperities. This failure appears to be a function of surface temperature, which is influenced by coefficient of friction, surface loading, sliding velocity, and rate of heat dissipation. Critical sliding velocity may therefore be changed by any factor affecting either the rate of heat generation or its dissipation.

The values of coefficient of friction obtained for a rubbed film of pure stearic acid on steel fall on a straight line between 0.09 at the lowest sliding velocity and 0.07 at the highest sliding velocity. For the rubbed films, friction values are lower than in the case of the solutions and lubrication was completely effective in preventing surface failure. The low friction values of pure stearic acid are probably a result of a relatively thick film of oriented crystals establishing a surface. This crystal orientation is a result of rubbing of the stearic acid film (reference 6). The molecular layer of stearic acid immediately adjacent to the steel surface reacts to form the metallic soap. The subsequent crystalline layers that may be formed in depth filling the microscopic recesses in the surface could attain a degree of orientation that probably would not be realized were the materials deposited without the physical rubbing of the surface (reference 6).

The practical implication that may be drawn from these data is that, for solutions of stearic acid in cetane, a breakdown velocity exists above which lubrication is no longer satisfactory. At the higher sliding velocities where lubrication failure occurs, increased stearic-acid concentration decreases the friction coefficient. The results presented in references 2 and 3 show that none of the temperature-active (extreme-pressure) additives studied provided effective lubrication at sliding velocities over 2000 feet per minute. Comparison of the data reported herein with those from references 2 and 3, which were obtained under the same conditions, indicates that stearic acid is more effective than several temperature-active additives in providing lubrication at high sliding velocities.

Fe_3O_4 film. - Data are presented in figure 5 showing the effect of sliding velocity on friction of steel surfaces having relatively thick (1000 A) prepared films of Fe_3O_4 when lubricated with stearic acid and cetane. For purposes of comparison, the curve for a dry Fe_3O_4 film (taken from reference 13) is presented. Figure 5 also presents data obtained with pure cetane, with solutions containing different concentrations of stearic acid in cetane, and with a rubbed film of pure stearic acid.

At the high sliding velocities, the friction obtained with a dry Fe_3O_4 film is in a range that compares favorably with some boundary lubricants. In contrast with the friction curve for cetane on clean steel, it should be noted that with pure cetane as the lubricant on Fe_3O_4, there is no apparent increase in friction with increased sliding velocity. This comparison indicates that the Fe_3O_4 film is effective in supplementing cetane to provide lubrication for steel surfaces.

In experiments using 0.1-percent stearic acid in cetane on Fe_3O_4, inconsistent friction data indicated that the lubrication was marginal. The data scatter was such that a curve could not be drawn through the points for the 0.1-percent solution; however, they all fell between the pure cetane curve and the curve obtained for the 0.5-percent stearic acid in cetane. These data indicate that concentrations greater than 0.1-percent are necessary to obtain completely effective lubrication under these conditions although Fe_3O_4 prevented surface welding. Data obtained with 0.5-percent stearic acid in cetane formed a smooth curve over the entire sliding-velocity range. No tendency toward surface failure was evident for the 0.5-percent solution even at the very high sliding velocities. In the high-sliding-velocity range (5000 ft/min and higher), there is very little difference between the friction obtained with the lubricated and unlubricated Fe_3O_4 films. This result might indicate that lubrication at the high sliding velocities is solely a function of the Fe_3O_4 film; however, it would then be expected that upon lubrication failure of the stearic acid-cetane solutions at 3000 feet per minute the curves for dry Fe_3O_4 and for 0.5-percent stearic acid in cetane on Fe_3O_4 would become coincident in the range from 3000 to 5000 feet per minute. Such a trend was not observed.

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The curve obtained when pure stearic acid (fig. 5) rubbed on the Fe_3O_4 film was used in the friction experiments shows lubrication to be completely effective over the entire range of sliding velocities. The friction obtained using 0.5-percent stearic-acid solutions approaches values for the pure stearic acid much more closely for the Fe_3O_4 film (fig. 5) than for the clean steel surfaces (fig. 4). The friction values for the two lubricants are within experimental error except in the very low range of sliding velocities between 75 and 500 feet per minute where the results for the pure stearic acid do not follow the downward trend with increasing sliding velocities common to other lubricants in this velocity range. Examination of the worn surface of rider specimens that had been lubricated with pure stearic acid, however, showed that the wear was significantly less than that obtained with stearic-acid solutions. It is assumed that these friction and wear data are dependent on film thickness and crystal orientation of the stearic acid as in the case of clean steel. In contrast, lubrication effectiveness of the stearic acid - cetane solutions is believed to be almost entirely due to the reaction film of metallic soap formed on the surface, as is indicated in reference 5 to be the lubrication mechanism of stearic acid on copper.

Fe_2O_3 film. - The presence of Fe_2O_3 films on steel surfaces formed in operation of lubricated sliders is normally associated with surface failure, as shown in reference 14. A simple experiment effectively demonstrated a factor that may be of importance in considering the possible use of stearic acid as an additive. Reduction of Fe_2O_3 to Fe_3O_4 was shown by mixing powdered Fe_2O_3 with stearic acid in a beaker and heating the resulting paste to a temperature where there was vaporization of the lubricant. Under these conditions, the color of the heated mass turned to black and the oxide became magnetic; both of these factors indicate that Fe_2O_3 was reduced to Fe_3O_4. Reduction of Fe_2O_3 by oxidation of stearic acid at temperatures above 266° F (130° C) may be a factor of importance in the use of stearic acid as an additive. The presence of stearic acid would therefore minimize the continued formation of Fe_2O_3 with its deleterious effects.

The effect of sliding velocity on friction of steel surfaces having prepared films of Fe_2O_3 lubricated with stearic acid and cetane is shown in figure 6. For the purpose of comparison, the figure includes a curve taken from reference 13 showing the friction properties of the unlubricated film of Fe_2O_3 formed on a

steel surface. The other data presented are those obtained with pure cetane on Fe_2O_3, with 0.1-percent stearic acid in cetane on Fe_2O_3, with 0.5-percent stearic acid in cetane on Fe_2O_3, and with a rubbed film of pure stearic acid on Fe_2O_3.

Lubrication of Fe_2O_3 with pure cetane was somewhat effective in the range of sliding velocities below 500 feet per minute regarding friction (fig. 6) and surface-failure tendencies. By comparison, cetane on Fe_3O_4 (fig. 5) provided effective lubrication over the entire range of sliding velocities with a constant friction coefficient of 0.15. The initial low values (fig. 6) and smooth sliding may have resulted from roughness or surface porosity of the Fe_2O_3 film. Roughness will effectively increase the energy of adhesion of the lubricant to the surface (reference 15). At sliding velocities of 1000 feet per minute and above, the sliding was not smooth, surface welding occurred, and occasionally there were violent fluctuations in friction-force measurements.

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For the range of sliding velocities below 3000 feet per minute, the curves for the 0.1-percent and the 0.5-percent solutions of stearic acid in cetane on Fe_2O_3 films were coincident and effective lubrication was obtained. At sliding velocities above 3000 feet per minute, surface failure occurred. The surface temperature at the critical velocity of 3000 feet per minute is believed to correspond to the transition temperature discussed in reference 16 for steel surfaces. The soaps of different acids would probably produce different critical velocities corresponding to the transition temperatures described in reference 16. Above a sliding velocity of 3000 feet per minute, the friction values for lubricant solutions on Fe_2O_3 approach those obtained for the pure cetane on Fe_2O_3. The curve for the lower concentration (fig. 6) breaks away from that for the higher concentration and becomes coincident with that obtained for the pure cetane at sliding velocities above 5000 feet per minute. With the higher concentration, the curve approaches that for the pure cetane on Fe_2O_3 film at the maximum sliding velocity reported.

Sliding velocity had very little effect on friction for the rubbed film of pure stearic acid on Fe_2O_3. The complete effectiveness of lubrication can again be attributed to film thickness and crystal orientation of the rubbed film.

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Effect of Surface Oxides

Stearic-acid solution (0.5-percent). - A comparison of the effects of different surface treatments for surfaces lubricated with a solution of 0.5-percent stearic acid in cetane is shown in figure 7. Lubrication failure occurred for the Fe_2O_3 film at essentially the same sliding velocity as for the clean steel specimens. Such coincident lubrication failure for the different surfaces suggests that a similar lubrication mechanism occurs with both Fe_2O_3 and with clean-steel films. The continued effective lubrication of the Fe_3O_4 film (fig. 7) at the highest sliding velocities is a result of other factors that will be subsequently discussed in the section on lubrication mechanism.

The results obtained at the lower sliding velocities confirm the work with a fatty acid reported in figure 2 of reference 17, which shows that the types of thick oxides formed on iron, copper, zinc, cadmium, and magnesium by different procedures did not have any pronounced effect on friction. At the higher sliding velocities, however, a very marked effect of the type of oxide (fig. 7) is apparent. The type of oxide film has a lesser effect on the friction values in the very low range of sliding velocities than in the range of velocities greater than the critical. At sliding velocities below the critical for the solutions, friction is somewhat lower where the prepared film is Fe_2O_3 than where the prepared film is Fe_3O_4. Study of the wear tracks indicates that the penetration of the film is less with Fe_2O_3 than with Fe_3O_4 or steel surfaces where effective lubrication was obtained.

Photographs are shown in figure 8 of sections of disk specimens that had been run with 0.5-percent stearic acid in cetane on a Fe_2O_3 film and on a Fe_3O_4 film. Visual observation after experiments indicated that lubrication failure had occurred on the specimen having the Fe_2O_3 film at high sliding velocities and did not occur except for high loads with the Fe_3O_4 film. Failure occurred with the beginning of "stick slip sliding", which accompanied the formation of minute welds on the contact surfaces.

The data presented in figure 9 show that the critical sliding velocity observed at approximately 3000 feet per minute was independent of oxide film (Fe_2O_3) thickness. It has previously been shown (fig. 6) that critical sliding velocity with a Fe_2O_3 film (1000 A thick) is independent of the concentration of stearic acid in cetane. The observation on effect of film thickness is based on a comparison of data obtained for a film thickness of 500 angstrom units (data points of fig. 9) with that obtained for a film thickness of 1000 angstrom units (dashed curve of fig. 9) using a stearic-acid concentration of 0.5-percent. At the lower sliding velocities, the values for the coefficient of friction fall within the experimental limits of the reference curve for the thicker (1000 A) Fe_2O_3 films. With films of 500 and 1000 angstrom units, lubrication effectiveness was independent of Fe_2O_3 film thickness.

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Pure stearic acid. - The effect of sliding velocity on friction of different surfaces lubricated with a rubbed film of pure stearic acid is shown in figure 10. At the minimum sliding velocity, no difference in friction is observed for the two prepared oxide films. The curves for the prepared Fe_3O_4 films and the clean steel surfaces (Fe_3O_4, 20 A) were nearly coincident, indicating again that the thickness of the oxide film is unimportant when effective lubrication is obtained. At all sliding velocities, friction is slightly lower for the Fe_2O_3 films than for the other surfaces. For all disk surfaces lubricated with the pure stearic acid, no wear tracks were visible without magnification when friction was 0.08 or less.

Load Effects

The effect of load on friction at various sliding velocities for steel having a film of Fe_3O_4 lubricated with 0.5-percent stearic acid in cetane is shown in figure 11. Data were obtained for loads from 169 to 1543 grams. Generally, friction increased with increasing loads. This characteristic was also observed with dry Fe_3O_4 films (reference 13). Marked increases in friction with load were observed in the load range from 169 to 269 grams and when lubrication failed. Failure (high friction and surface welding) was observed at loads above 769 grams with sliding velocities of 3000 feet per minute and higher. These data points were unstable because the surfaces were subjected to progressive failure at the time the data were taken.

A series of photomicrographs showing surfaces of disk specimens lubricated with 0.5-percent stearic acid in cetane after various conditions of sliding is shown in figure 12. Smooth wear tracks characteristic of effective lubrication are shown for clean steel and for a Fe_2O_3 film in figures 12(a) and 12(b), respectively. Figure 12(c) shows the welding that accompanied lubrication failure of an Fe_2O_3 film at high sliding velocity. Comparison of figures 12(b) and 12(d) shows that increased loads can also lead to drastic surface failure by mass surface welding. Comparison of figure 12(d) with figure 12(e) illustrates that Fe_3O_4 has greater load capacity than Fe_2O_3. With a 1017-gram load, failure of a Fe_3O_4 film did not occur until the sliding velocity had been increased to 3000 feet per minute (fig. 11); failure of a Fe_2O_3 film occurred at 1000 feet per minute with the same load (fig. 12(d)). Figure 12(e) shows some plastic flow of the surface. A greater amount of plastic flow is evident in figure 12(f), which shows a surface produced by more severe sliding conditions. On both edges and in the bottom of the wear tracks, material has flowed sufficiently to partly fill coarse finishing marks.

Lubrication Mechanism

The mechanism of lubrication by fatty-acid solutions can be described as follows: With very light loads, effective lubrication is provided by strongly physically adsorbed films of the additive. With slightly heavier loads, the adsorbed film is penetrated and lubrication is provided by the metallic soap film. The metallic soap, with increased loads or velocities, softens or melts and under extreme conditions breaks down. After the melting point of the metallic soap has been reached, the soap breaks down and any further lubrication is dependent on the oxide. Results presented herein show that the oxide Fe_3O_4 prevented lubrication failures under extreme conditions. Studies of Fe_3O_4 described in reference 18 (p. 745) show that under conditions of severe load magnetite crystals break into pieces and that the fragments have the form of little plates that separate from the crystal parallel to the octahedral faces. It is also reported in reference 18 that the Fe_3O_4 does not exhibit any tendency toward plastic deformation. It is possible that adsorbed films and metallic soaps may have formed on the surfaces of these fragments. Study of some of the wear tracks in a number of the runs have shown that the Fe_3O_4 has a tendency to flow in tracks where surface deformation had occurred. Because the

Fe_3O_4 is not a plastic material, it might reasonably be suggested that the crystals of the oxide were fragmented and that the resulting plates spread over the surface. These plates may lubricate in a manner very similar to graphite. By contrast, Fe_2O_3 did not protect the surface under extreme sliding conditions and was completely scaled out of the contacting area when film rupture occurred.

When the oxides are formed at low temperatures, as was the case in these experiments, the adsorptive capacity is much greater than if formed at higher temperatures (reference 18, p. 808). If the lubricating additive penetrated the porous oxide film, it is probable that a reaction product was formed within the film. The oxide will then be comparable with the hard matrix of a bearing material and may function by extrusion of the fluid lubricant or reaction product according to the thin-metallic-film theory of lubrication of bearing materials. Typical of this type of material is the copper-lead bearing, where the low shear strength lead is extruded from within the hard copper matrix. Any tendency of the lubricating film, either the fluid, adsorbed additive, soap, or oxide film, to flow over the surface and prevent metallic contact is beneficial in providing effective lubrication.

SUMMARY OF RESULTS

Lubrication of clean steel and oxide-coated steel surfaces was studied using stearic acid as a lubricant additive in cetane and as a pure rubbed film. At sliding velocities between 75 and 7000 feet per minute with loads from 169 to 1543 grams, the following results were observed:

1. A prepared film of ferroso-ferric oxide Fe_3O_4 prevented lubrication failure with stearic acid and cetane at sliding velocities higher than 7000 feet per minute (269 gram load) and with loads to 769 grams at a sliding velocity of 6000 feet per minute. Friction decreased slightly with increased sliding velocity.

2. Lubrication with stearic acid as an additive in cetane was effective to sliding velocities of 3000 feet per minute with clean steel surfaces and surfaces coated with ferric oxide Fe_2O_3. The lubrication failures were probably caused by melting of the metallic soap formed by reaction of the stearic acid with the oxide films on the steel surfaces.

3. Thickness of the films of Fe_3O_4 on clean steel (approximately 20 A) and with the prepared oxide film (approximately 1000 A) influenced lubrication failure with stearic acid in cetane at high sliding velocities where the thicker film prevented failure. Friction values were duplicated with the two types of surface at sliding velocities below 3000 feet per minute. Thickness of Fe_2O_3 films did not influence friction data to any marked extent.

4. A rubbed film of pure stearic acid was effective in preventing lubrication failure on clean steel and oxide-coated surfaces with a load of 269 grams at sliding velocities above 7000 feet per minute.

Lewis Flight Propulsion Laboratory,
National Advisory Committee for Aeronautics,
Cleveland, Ohio, January 30, 1951.

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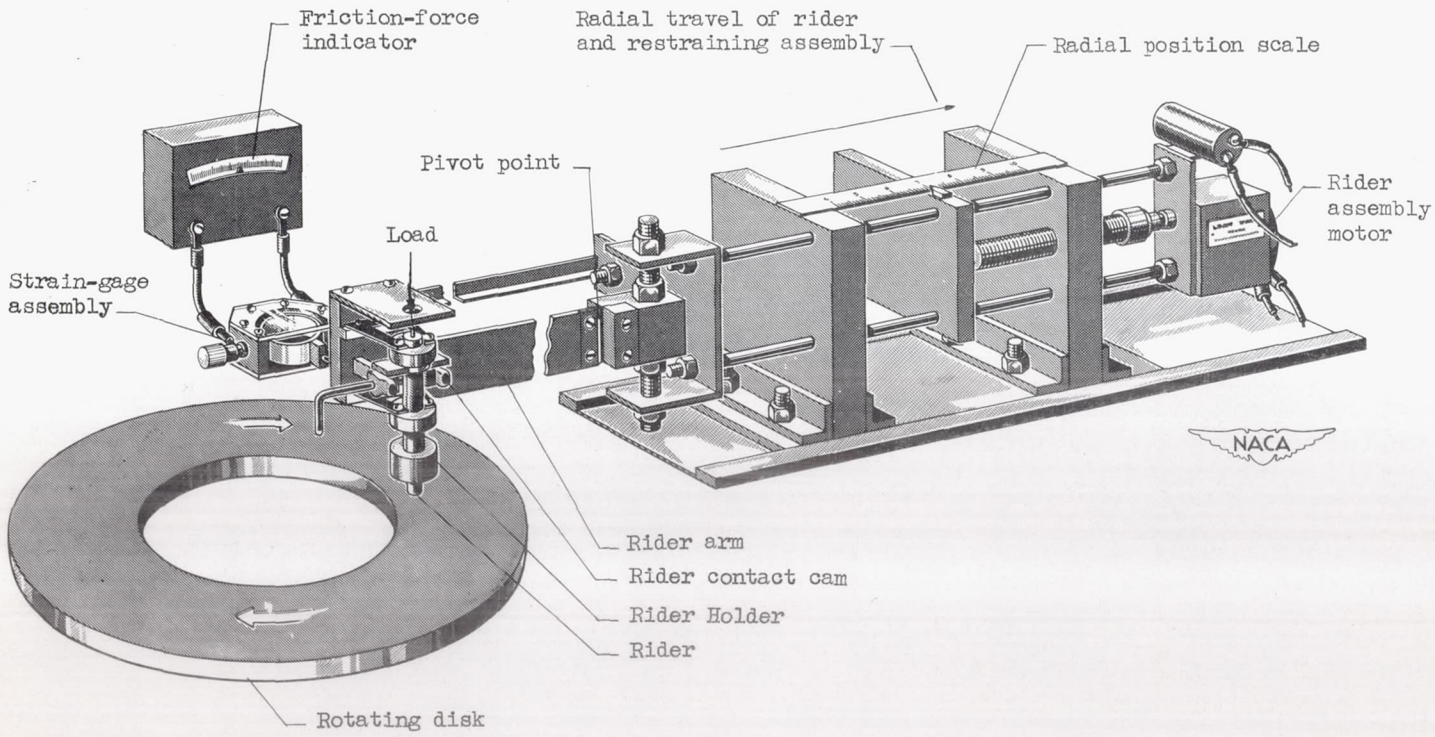


Figure 1. - Schematic diagram of sliding friction apparatus.

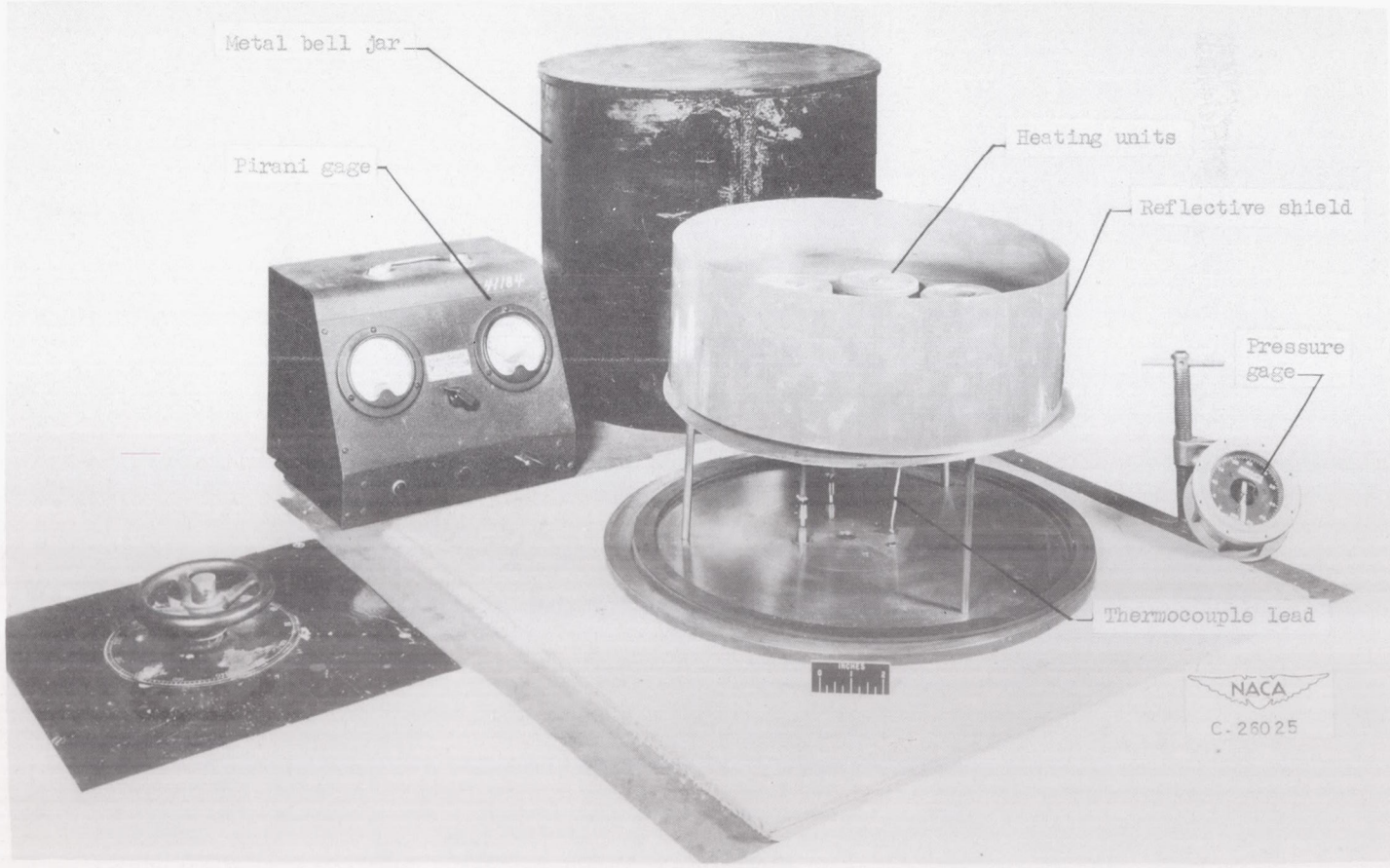
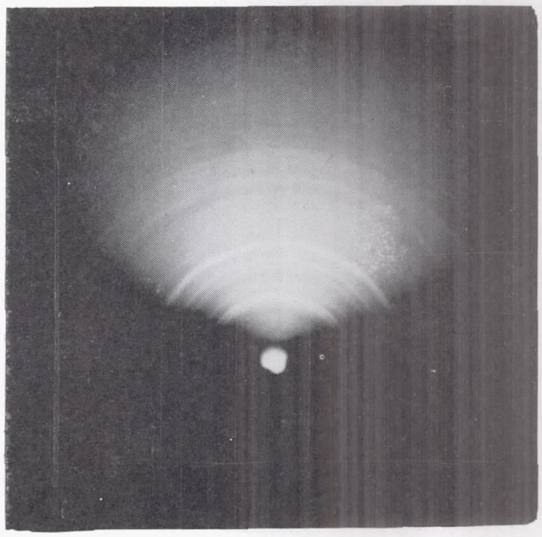
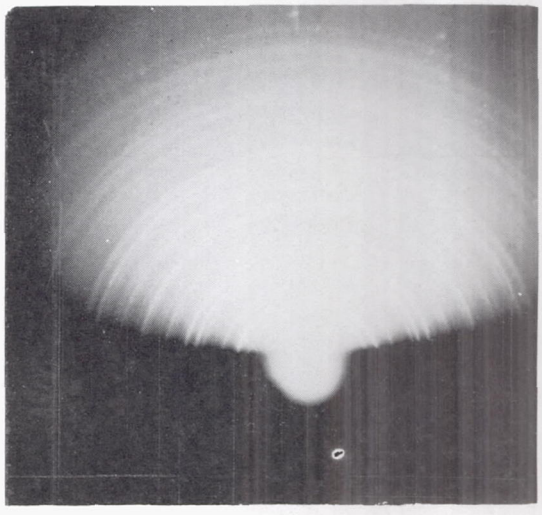


Figure 2. - Photograph of vacuum furnace used to form oxide films.



(a) Fe_2O_3.



(b) Fe_3O_4.

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Figure 3. - Electron diffraction patterns of oxide films.

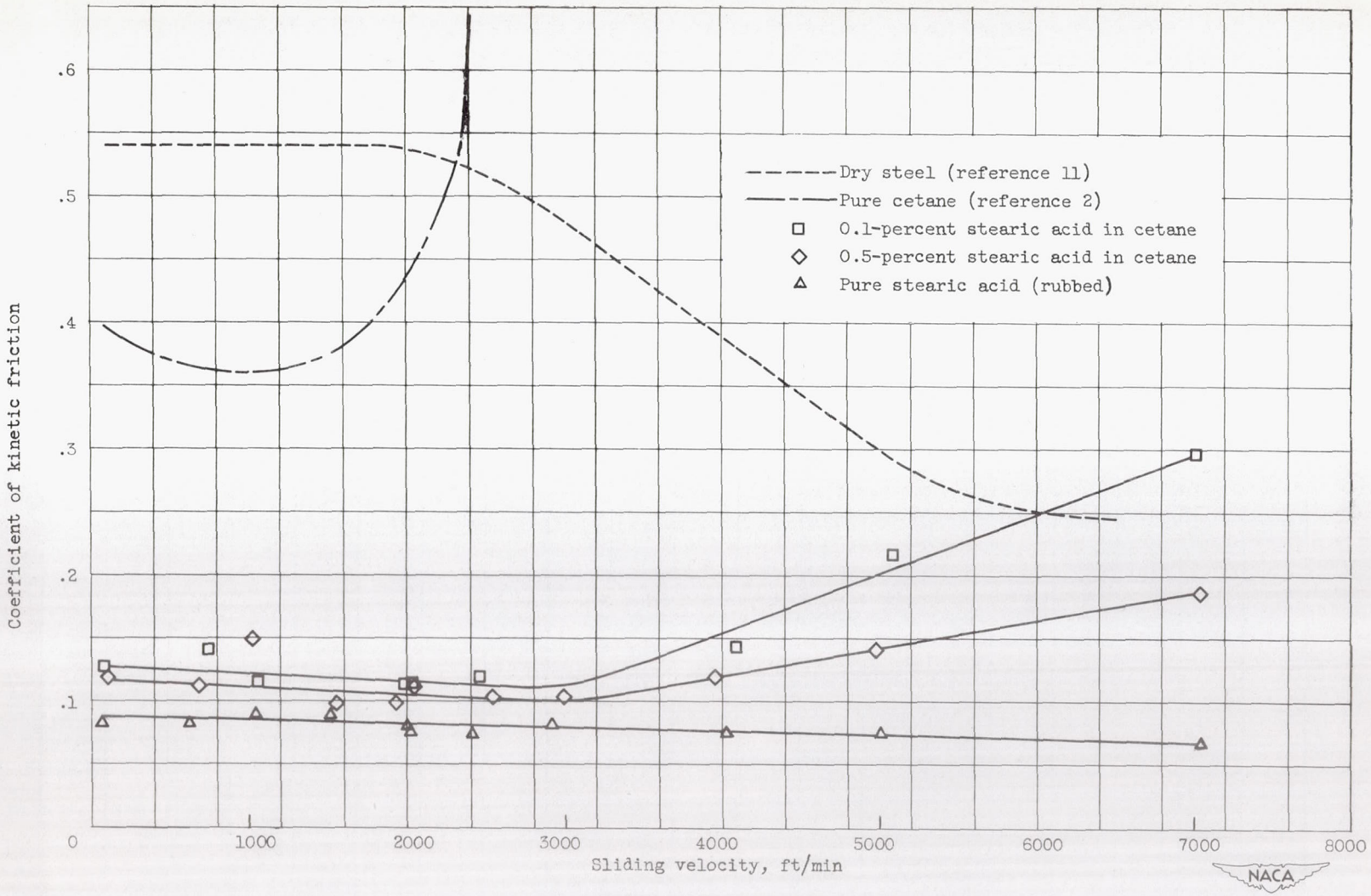


Figure 4. - Effect of sliding velocity on friction of steel surfaces lubricated with stearic acid and cetane. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.).

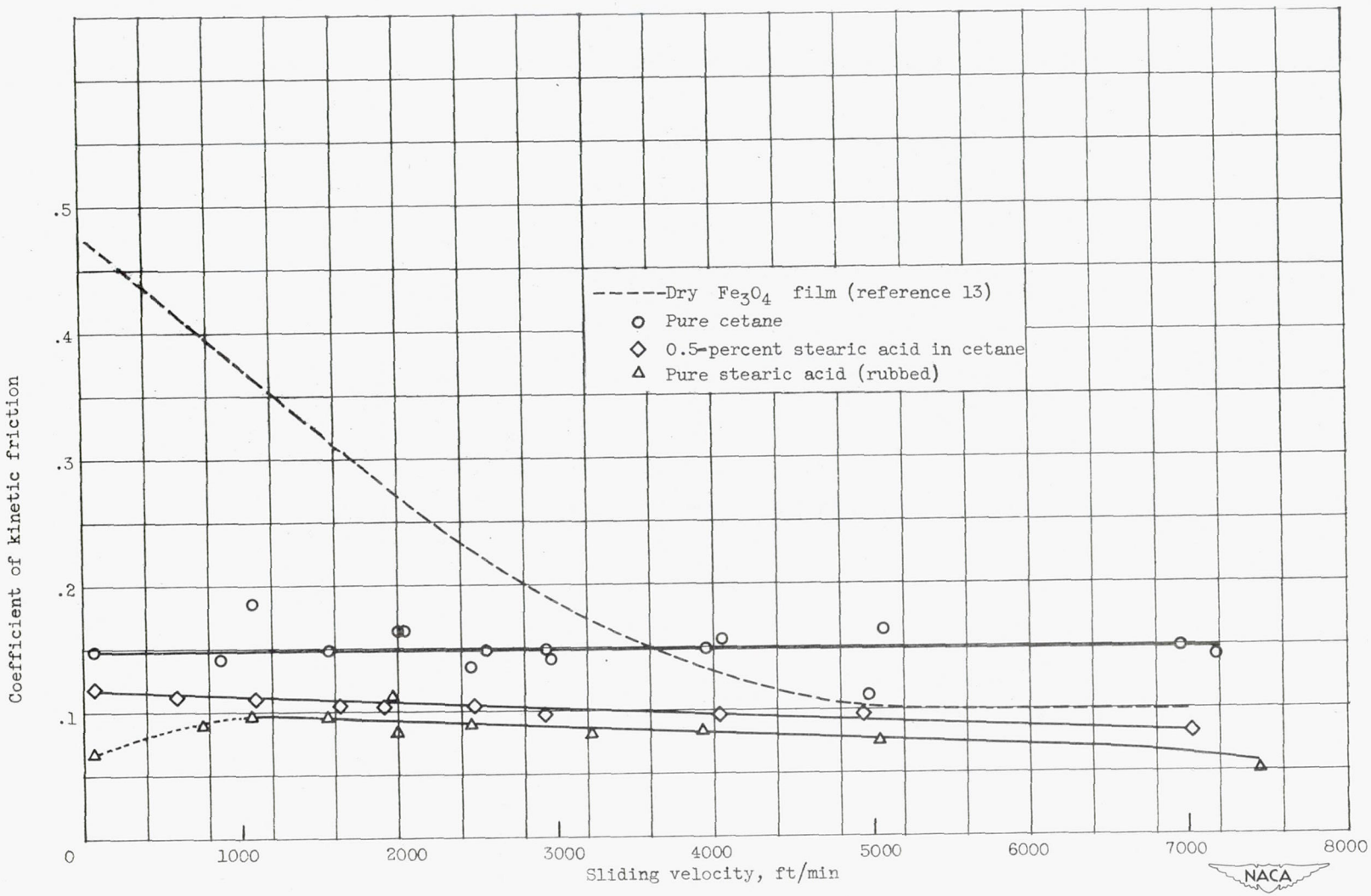


Figure 5. - Effect of sliding velocity on friction of steel surfaces having prepared films of Fe_3O_4 (1000 A thick) lubricated with stearic acid and cetane. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.).

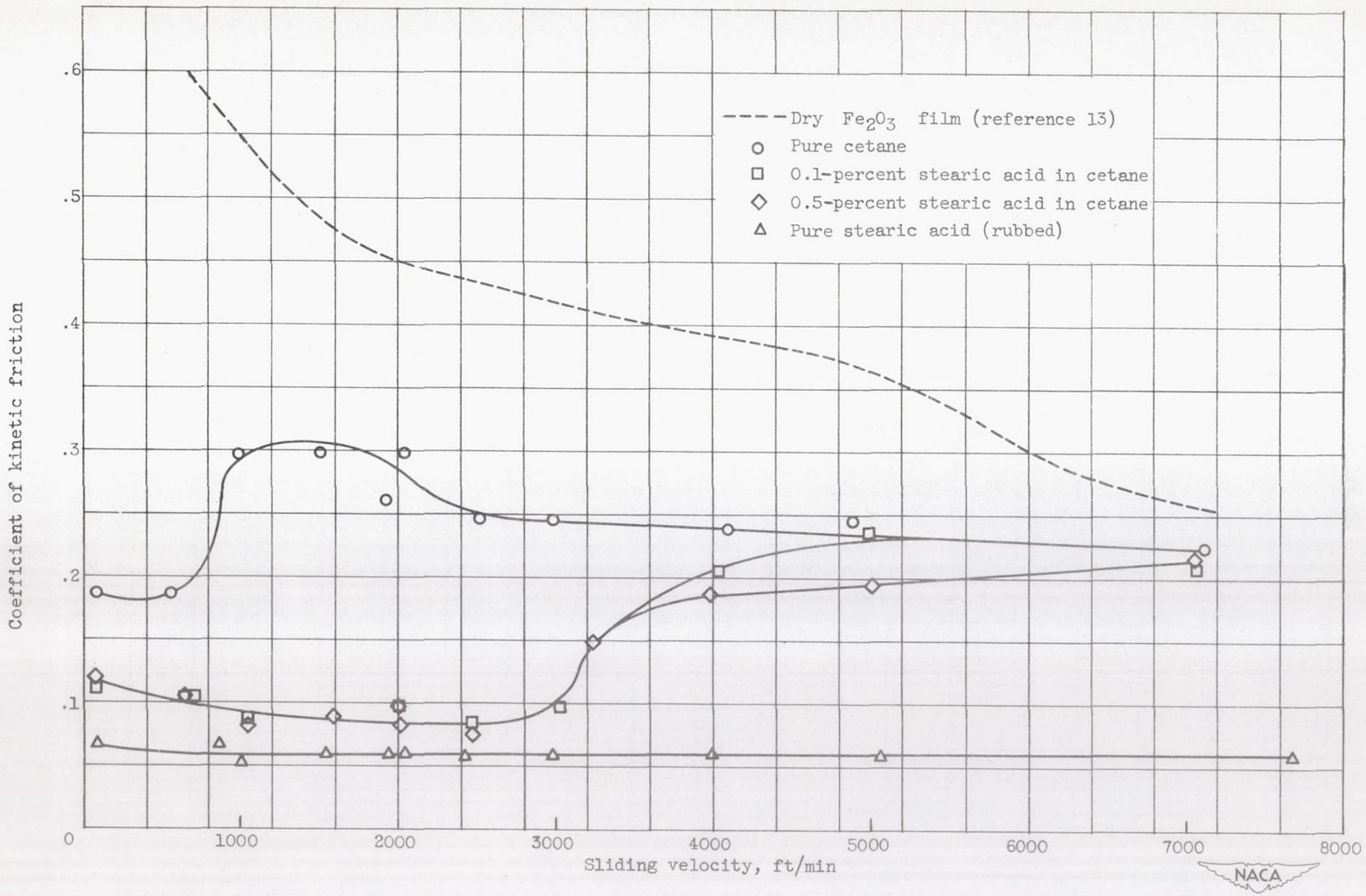


Figure 6. - Effect of sliding velocity on friction of steel surfaces having prepared films of Fe_2O_3 (1000 A thick) lubricated with stearic acid and cetane. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.).

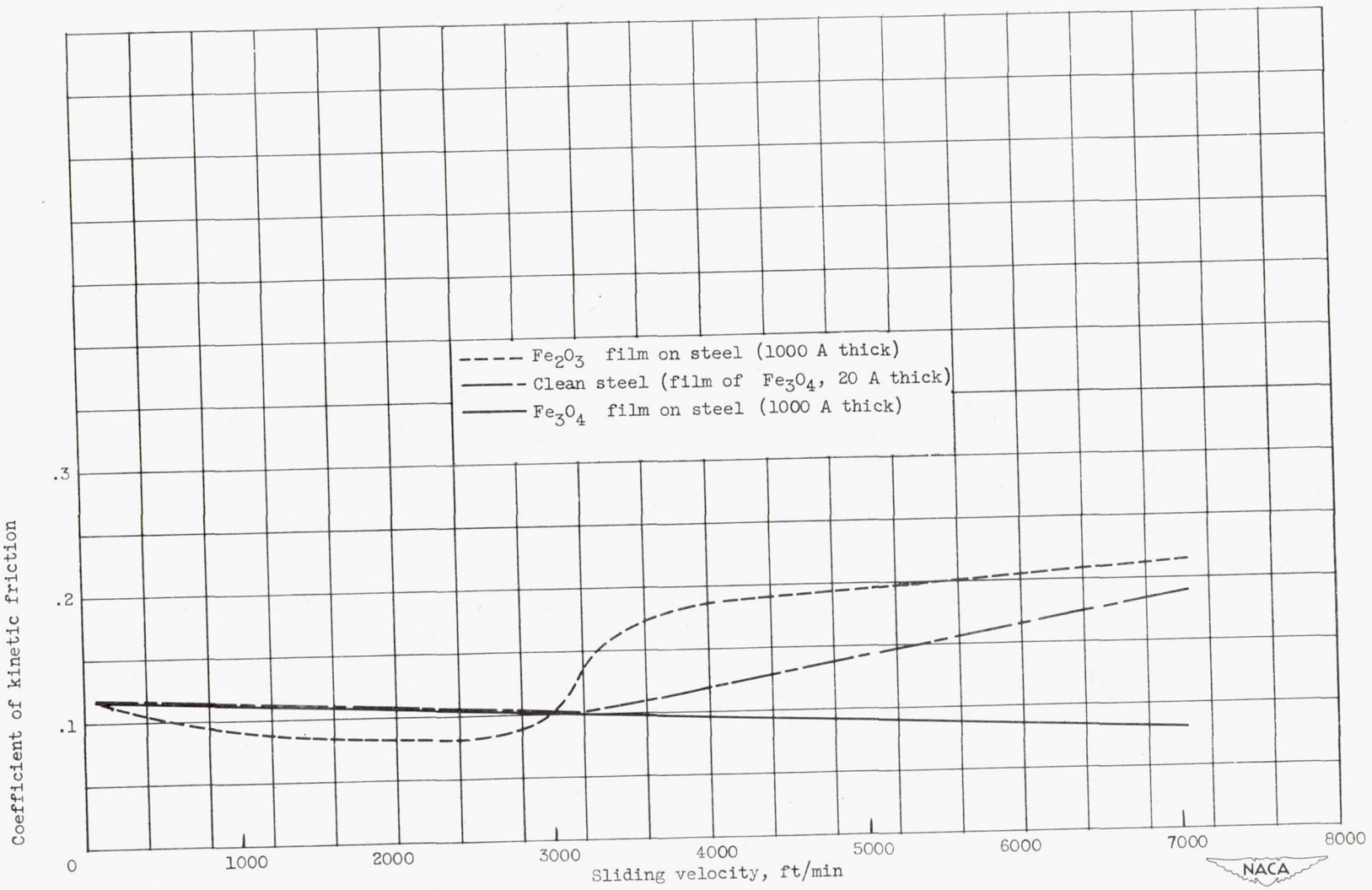


Figure 7. - Comparison of effect of various surface treatments on friction of surfaces lubricated with 0.5-percent stearic acid in cetane. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.).

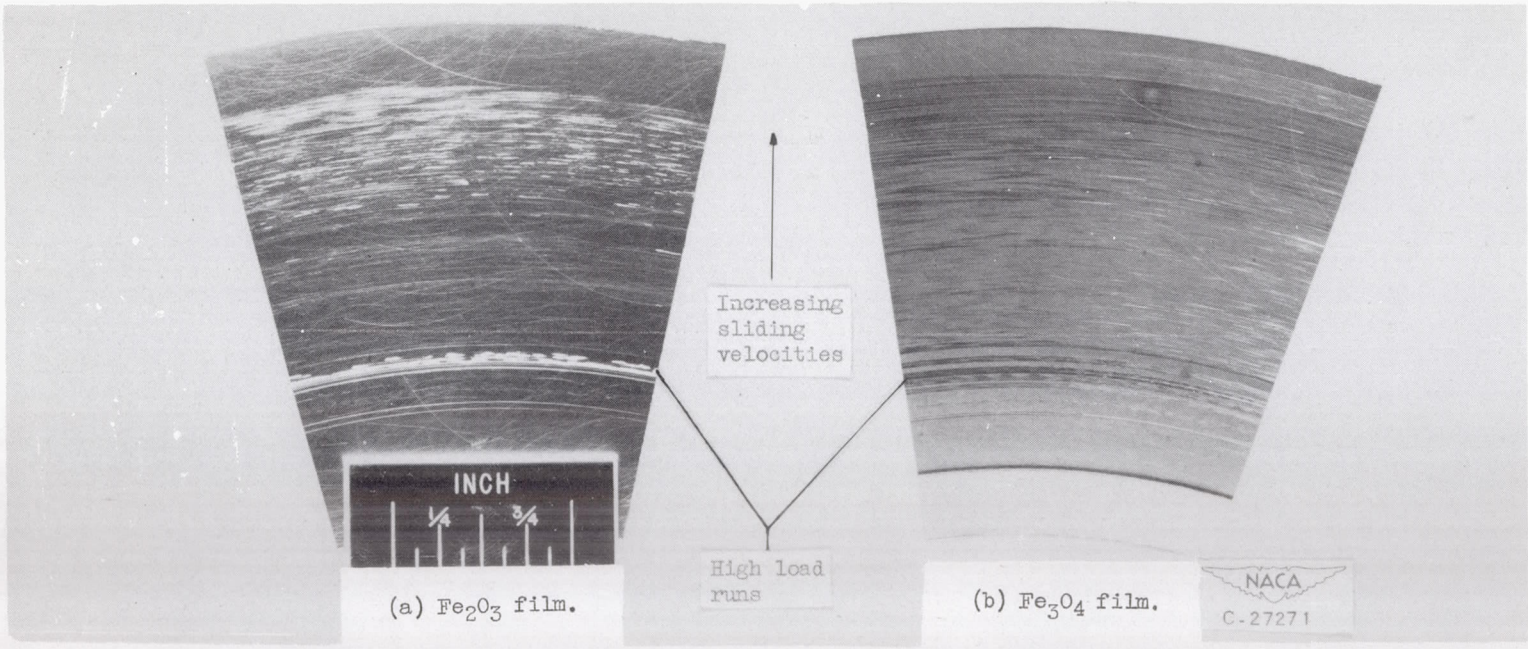


Figure 8. - Photographs, after friction experiments, of sectors of disk surfaces having oxide films. Lubricated with 0.5 percent stearic acid in cetane. Sliding velocities, 75 to 8000 feet per minute; loads, 269 to 1017 grams (initial Hertz surface stress, 126,000 to 194,000 lb/sq in.).

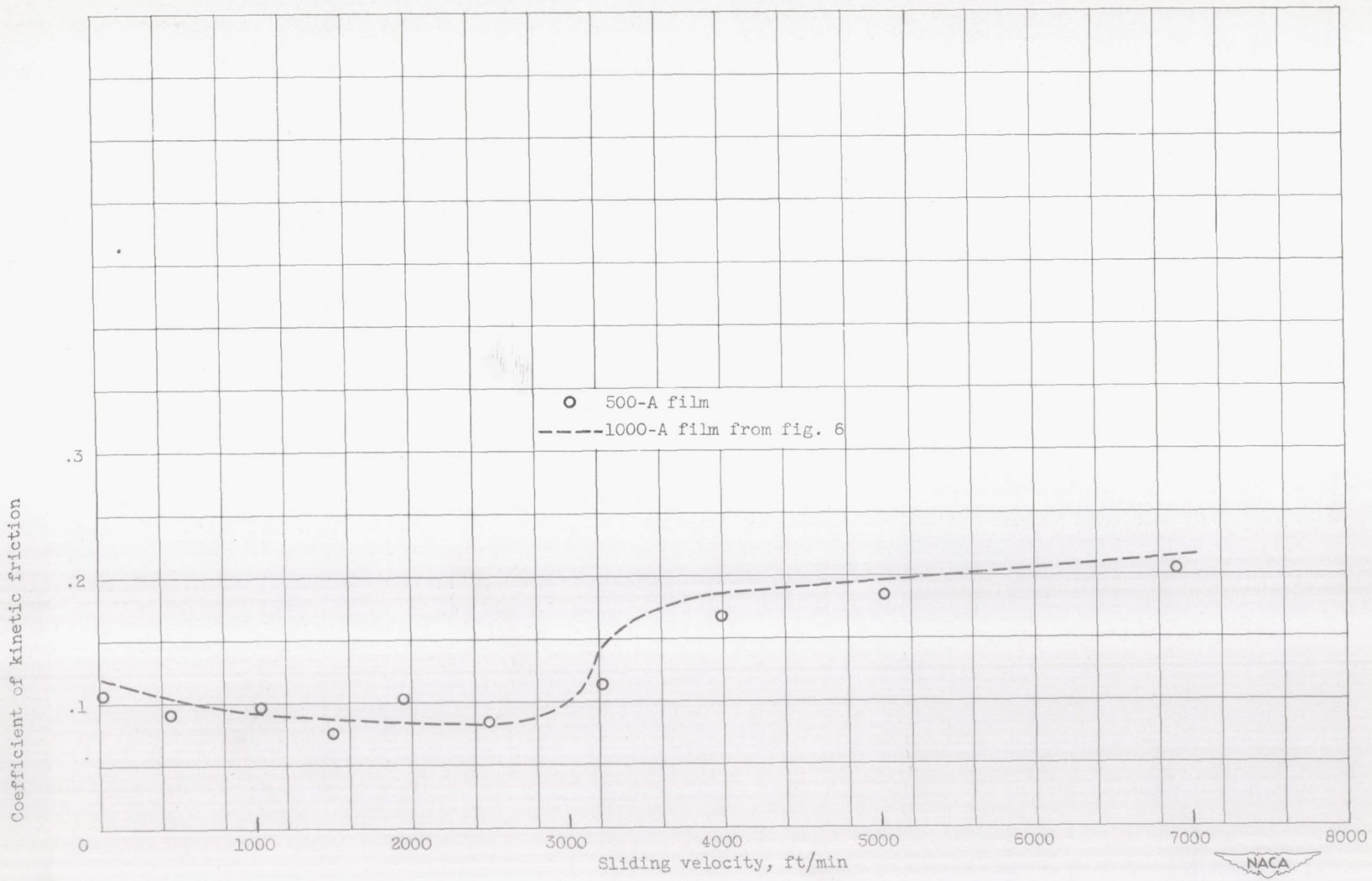


Figure 9. - Effect of sliding velocity on friction of steel surfaces having prepared surface films of Fe_2O_3 and lubricated with 0.5 percent stearic acid in cetane. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.).

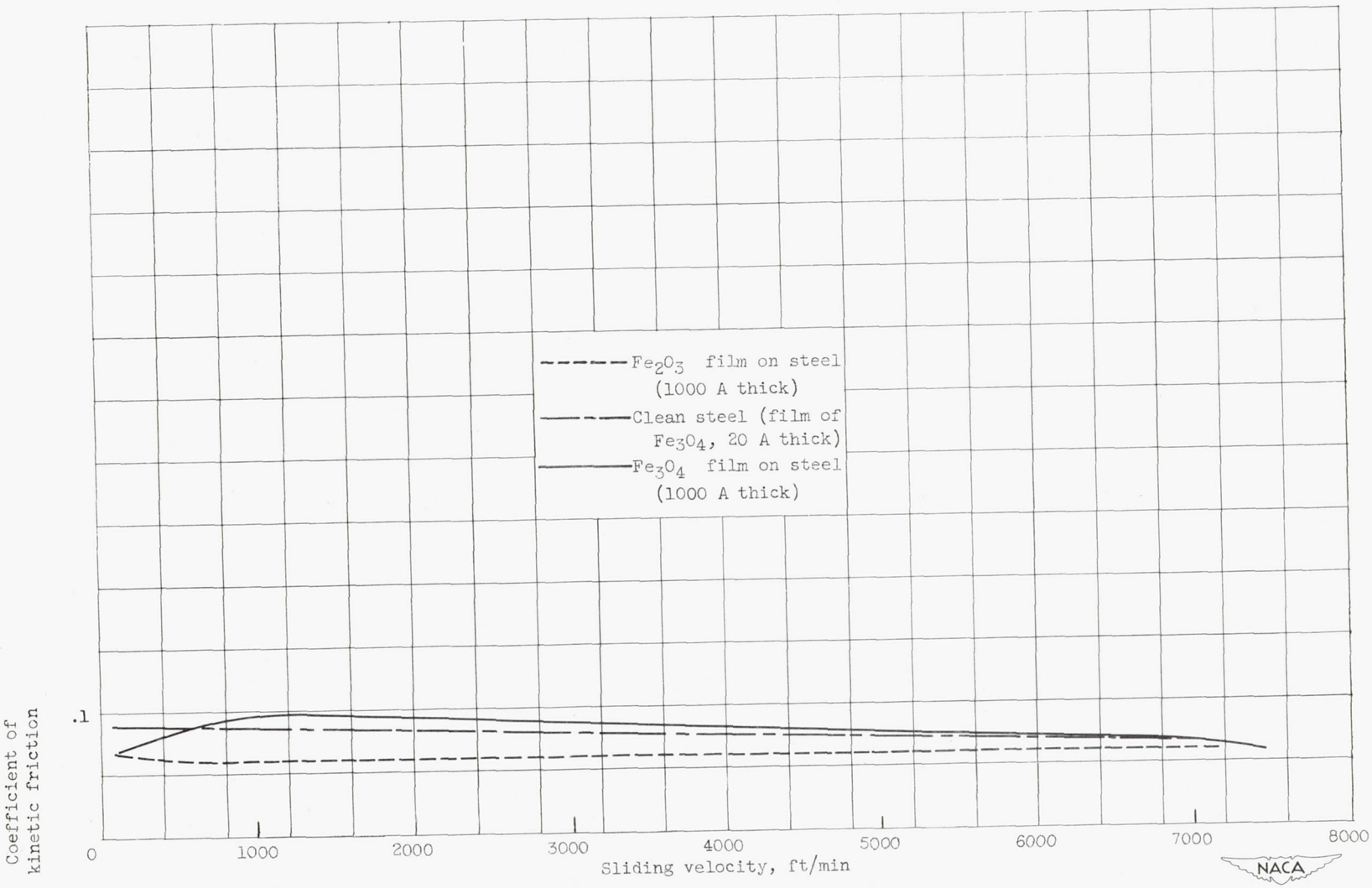


Figure 10. - Comparison of effect of various surface treatments on friction of surfaces lubricated with rubbed film of stearic acid. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.).

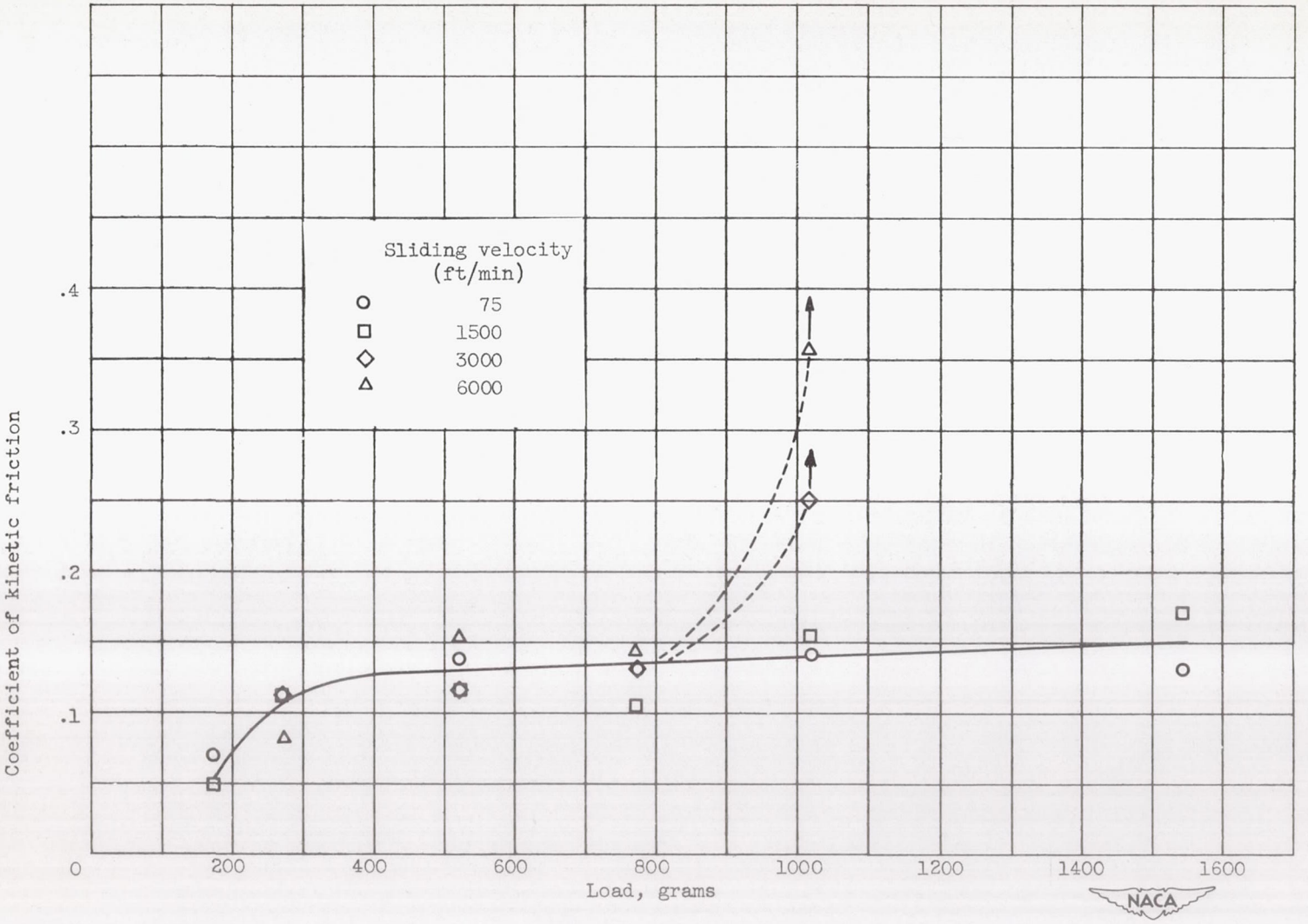
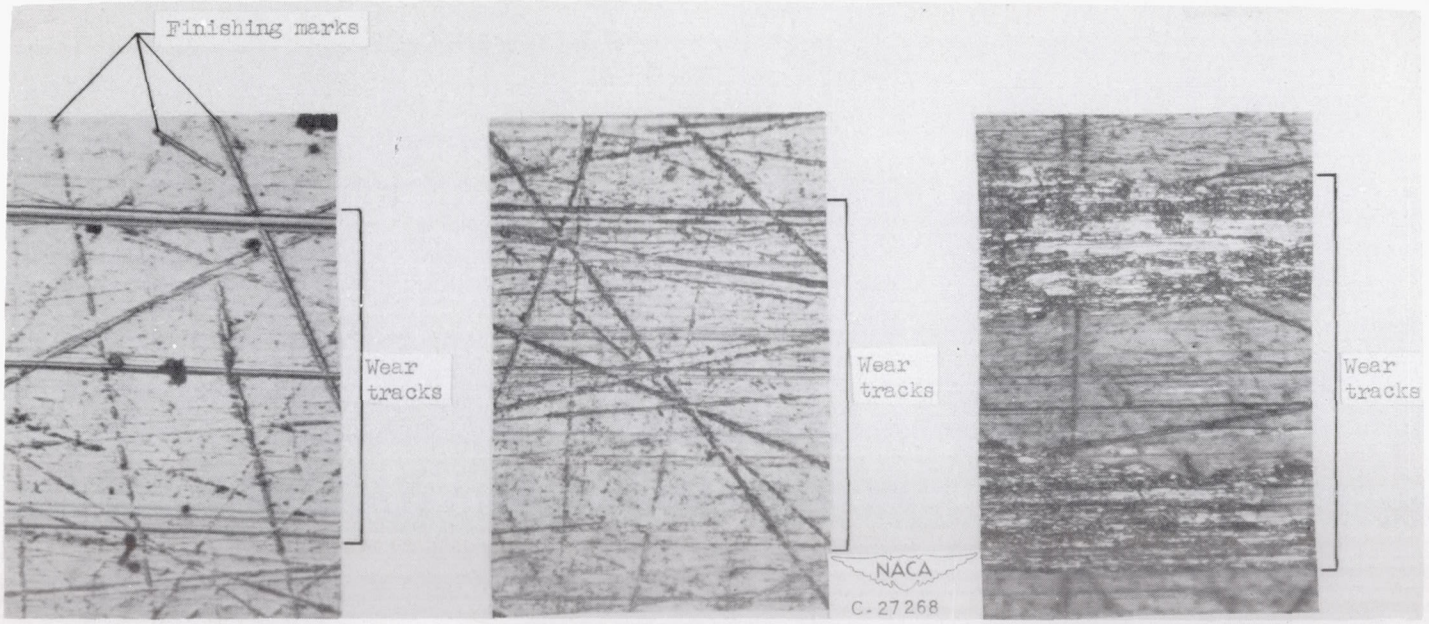


Figure 11. - Effect of load on friction for steel with Fe_3O_4 film (1000 A thick) lubricated with 0.5-percent stearic acid in cetane.

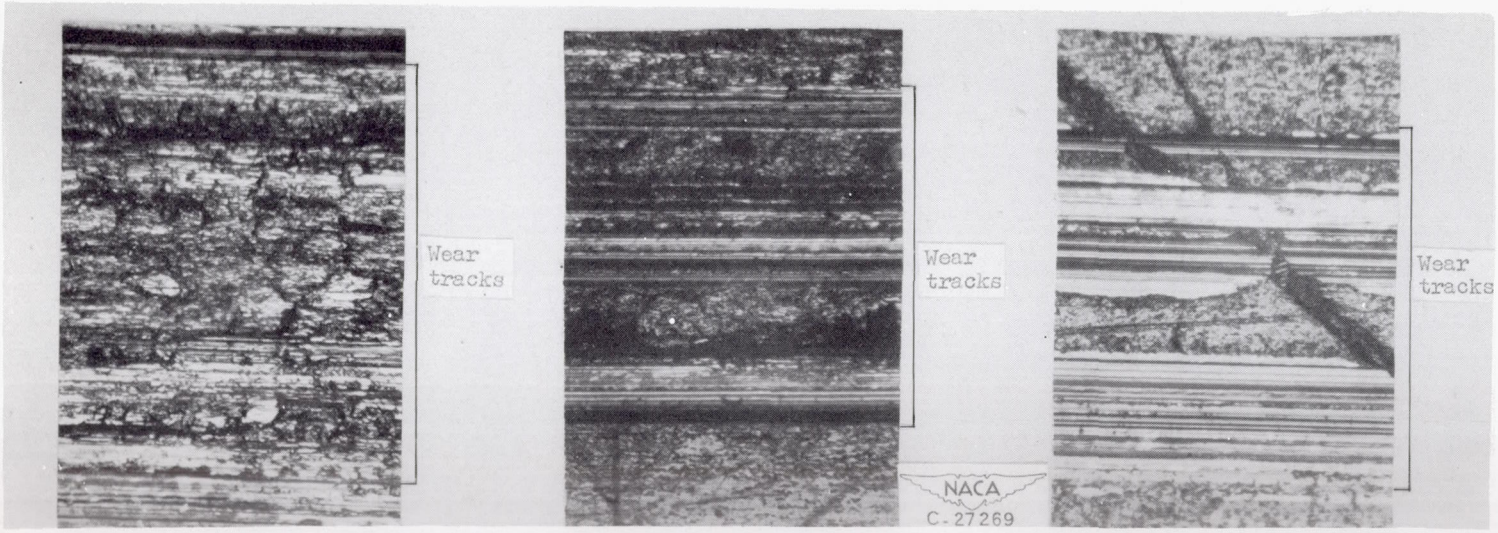


(a) Effective lubrication with untreated steel. Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.); sliding velocity, 2000 feet per minute.

(b) Effective lubrication with film of Fe_2O_3 (1000 A thick). Load, 269 grams initial Hertz surface stress, 126,000 lb/sq in.); sliding velocity, 2000 feet per minute.

(c) Lubrication failure resulting from high sliding velocity with film of Fe_2O_3 (1000 A thick). Load, 269 grams (initial Hertz surface stress, 126,000 lb/sq in.); sliding velocity, above 3000 feet per minute.

Figure 12. - Photomicrographs of friction-specimen surfaces after sliding contact under various conditions of lubrication by 0.5 percent stearic acid in cetane. X150.



(d) Lubrication failure resulting from high loading with film of Fe_2O_3 (1000 A thick). Load, 1017 grams (initial Hertz surface stress, 194,000 lb/sq in.); sliding velocity, 1000 feet per minute.

(e) Effective lubrication with film of Fe_3O_4 (1000 A thick). Load, 1017 grams (initial Hertz surface stress, 194,000 lb/sq in.); sliding velocity, 1000 feet per minute.

(f) Effective lubrication with film of Fe_3O_4 (1000 A thick). Load, 769 grams (initial Hertz surface stress, 194,000 lb/sq in.); sliding velocity, 3000 feet per minute.

Figure 12. - Concluded. Photomicrographs of friction-specimen surfaces after sliding contact under various conditions of lubrication by 0.5 percent stearic acid in cetane. X150.